Test and Measurement Coalition

RoHS Scope Review of Category 9 Products

Hexavalent Chromium

Abstract

Test and Measurement (T&M) products represent a very high performance subset of the monitoring and control product category. These products are characterized by extremely sensitive electrical systems that must perform at levels well beyond the conventional electrical products they test. This paper describes why electromagnetic shielding is necessitated by the extreme sensitivity of test and measurement equipment to electromagnetic interference (EMI) as well as the challenge of meeting increasingly stringent regulatory EMI requirements under consideration in the European Union. This paper also describes two phases of testing with inconclusive results thus far, in the ability of substitute coatings to control the suppression of EMI.

Electromagnetic shielding is an integral part of high performance instruments, playing a significant electrical role in the overall performance of T&M products. By documenting test results that link available finish materials to electromagnetic performance, we expect to illustrate the importance of shielding characteristics in T&M products.

Hexavalent Chromate Conversion Coating (HCCC) has served as a standard and robust materials finish for various metals, particularly aluminium and steel, to assure adequate and appropriate electromagnetic shielding as required by the EMC Directive, 89\336\EEC, as amended. To successfully replace HCCC, complex and time consuming testing and evaluations are required to ensure sustained and repeatable electrical performance in test and measurement applications and use environments. These include at minimum, material finish characterizations and electromagnetic performance testing across a broad range of test and measurement products. As of this writing, there are no representative industry studies, testing, or benchmarks for the replacement of HCCC in T&M products. To study the feasibility of replacing HCCC, the T&M sector companies have conducted some preliminary testing of replacement finishes, the results of which are inconclusive. Furthermore the tests were done only on some sample materials used in some of the parts of the final product. The tests are in no way representative of the effects on a real product or even a part of a product.

Based on results to date, the T&M sector requests an exemption to the RoHS Directive for the use of Hexavalent Chromium Conversion Coating in test and measurement equipment as well as a phase-in period to bring products into compliance once a suitable alternative has been proven.

The exemption is required for a period of four years from the inclusion of T&M products in the scope of RoHS. This will provide the time to develop a suitable alternative. After four years the exemption can be reviewed with a view to granting an extension or starting the phase-in period.

A transition phase-in period of four years from inclusion of T&M products in the scope of RoHS to substitute any other use of hexavalent chrome in our products. This period is considered realistic to conduct supplier surveys and replace any instances found in other applications of hexavalent chrome.

1.0 General

Hexavalent Chromium Conversion Coating is used in Test and measurement equipment as a finish on metal surfaces. The conversion coating consists partially of hexavalent chromium (Cr^{6+}) in a very thin layer, on the order of 2 μ m thick. It is calculated that members of the T&M Coalition, who comprise roughly 60% of the T&M sector, place approximately 6.81 Kg per year of Hexavalent Chromium on the EU market. This material has been used for many years in a number of industry sectors for its distinct and significant advantages described below.

1.1 Technical Characteristics

HCCC provides the following characteristics when applied to metal surfaces:

- Corrosion retardation when used under supplementary organic finishes or films
- Corrosion retardation without materially changing electrical resistivity
- Improved adhesion for organic finishes
- Mild wear resistance
- Enhanced drawing or forming characteristics
- Decorative or cosmetic purposes, when coloured or dyed

The simplicity of the basic process makes HCCC easy to apply. HCCC also provides a "self healing" surface and does not affect fatigue resistance of the material.

1.2 Use of Hexavalent Chromium in Test and Measurement Equipment

Hexavalent Chromate Conversion Coatings are critical to the test and measurement equipment industry as surface treatments specifically used to protect metal parts from corrosion, and to provide an electrical path for ground currents and electromagnetic shielding. T&M equipment is expected to operate properly under extreme conditions and these coatings have proven to be extremely effective in meeting these requirements as documented by multiple independent peer review publications. An additional benefit is that

they are self healing, that is, they continue to protect the metal surface even if scratched as the chrome layer flows back and re-passivates the area.

Test and measurement equipment not using this or a similar corrosion protective coating can result in rapid and severe corrosion. Such corrosion leads to poor control of radiated and conducted electromagnetic interference (EMI) due to loss of consistent electrical continuity in the electromagnetic shielding, impacting instrument performance. This affect can compromise the ability of T&M equipment to meet regulatory requirements intended to protect the customer and surrounding equipment from excess EMI.

Figure 1 and 3 show several representative T&M instruments that meet current regulatory requirements and utilize HCCC aluminium shielding panels.





Figure 1 Figure 2

Tektronix TDS 5104B with and without plastic shell installed





Figure 3 Figure 4

Tektronix TDS 7404B with and without plastic shell installed

HCCC is an integral element in the design of continuous and reliable electromagnetic shielding that meets the high performance product requirements of test and measurement equipment needed to control radiated and conducted electromagnetic interference (EMI), while providing a cosmetically appealing surface finish over an extended product lifetime.

1.3 Trends

T&M instruments have a long life (10+ years) and HCCC has been a proven coating over many years in fighting corrosion and enabling EMI performance while maintaining a corrosion-free cosmetic finish. Due to the proven performance of HCCC over many years of use, the ability of potential substitute materials to meet current specifications and customer acceptance criteria must be carefully substantiated by verification testing. The approval process for substitute coatings will require significant effort over a period of time. Previous work to qualify alternates to HCCC has focused on corrosion protection alone, without taking into account the needs of the T&M industry. To the best of our knowledge, the testing being performed by T&M Coalition members is the only effort that has been made to determine the suitability of potential substitutes for HCCC to meet the long term EMI performance requirements important to the T&M product sector.

As stated in the Third Report on RoHS Compliant Alternatives to Hexavalent Chromium for Treatment of Steel and Aluminum, "...the metal finishing industry generally, is going through a period of rapid change and development. Consequently, we can expect further processes and process developments to be launched, in the next year or two at least." This trend is upending the supply chain as new suppliers and products must be qualified. Over time, suppliers will settle on processes and products that meet their customer's needs which will be based on validation of performance.

2.0 Substitutes

A number of surface treatments for steel and aluminum have been cited in referenced papers.^{1,2} According to these sources, investigative testing of the following finishes has been completed using steel and various aluminum alloy materials:

Henkel Alodine 1200(S) (Chromate or Hexavalent Chromium)

MacDermid ELV Blue (Product Number: IP74330)

MacDermid PK3 Blue

Chematall Oxsilan Al-0500

Henkel Alodine 4595

APS Chemicals Surtec 650 (TCP-HF)

Bi-K Aklimate

Boeing Boegel Sol-gel (Advanced Chemistry and Technology, Inc as AC-131)

Bent OXSiLAN® AL-0500

² ESTCP Phase I report on Non-Chromate Aluminum Pretreatments, Aug 2003

¹ RoHS and WEEE Specialists (NZ) Ltd, 25 October 2005

Fortune Chemical Co X-It PreKoteTM
Henkel Surface Technologies Alodine 5200® and Alodine® 5700
MacDermid Chemidize® 727ND
NAVAIR Trivalent Chromium Pretreatment (TCP)
Sanchem Safegard 7000

2.1 Impact of Substitution

The list of possible substitutes to HCCC continues to grow. Each of these substitutes has benefits and disadvantages that need to be fully explored. The effects of "drop-in" substitution across the range of test and measurement products in this category is currently being explored but requires more time to complete ongoing and planned testing.

Any HCCC substitute suitable for test and measurement applications must enable the control of radiated EMI and meet the need for low-weight electromagnetic shielding over an extended life in potentially hazardous and extreme environments. Four reasons, described below, warrant a comprehensive and robust investigation of substitutes to HCCC as a surface coating in test and measurement equipment.

1. Test and measurement equipment has unique challenges in controlling radiated EMI. The very high frequency signal generation and acquisition technology used by test and measurement equipment coupled with customer expectation of light weight and portability makes controlling radiated EMI by aluminum shielding a serious engineering challenge. The low electrical resistance of HCCC makes it the preferred coating for a continuous electromagnetic shield that mitigates the effects of EMI. Our DC resistance testing confirms that HCCC has a lower electrical resistance than several other substitute coatings. This suggests that HCCC may be more effective than other substitute coatings in controlling EMI emissions when used on aluminum shields (although the shielding effectiveness tests covered in this report gave ambiguous results). Higher impedance mechanical connections caused by HCCC substitute aluminum coatings may cause inconsistent electrical ground current patterns, forming antennas that radiate higher energy electromagnetic emissions. This is caused by ground currents having to flow around poor connections. This effect raises the expectation that emissions will increase and become more variable with increased corrosion and may be better illustrated by actual product testing.

Today, some complex products are only capable of achieving the thinnest of margins with respect to EMC regulatory limits. Small changes can alter a response curve, evaporating the margin and shifting the curve to nonconforming.

2. A light weight but highly effective EMI shield is also necessitated by the extreme sensitivity of test and measurement equipment to external EMI. This sensitivity is due to the need to maintain high 'signal to noise' ratios in the signal acquisition systems even when measuring extremely low level, high frequency signals. Note that these signal acquisition systems must be much more sensitive and operate at higher frequencies than the most sophisticated electronic equipment that they test.

- 3. Test and measurement equipment is typically used for ten years with many products being used for more than 20 years. HCCC has self-healing properties allowing it to reliably provide corrosion protection in harsh environments over long product lifetimes. This coating solution has proven itself in the field over decades and any substitute must be introduced with confidence that it will be comparably robust.
- 4. Construction of a continuous and effective electromagnetic shield is possible without HCCC by sacrificing light weight and small size. The use of zinc plated or galvanized steel, common in some sectors of the electronics industry, is not desirable for our customers who require lightweight, small size, and portability in field applications. Testing shows that plated steel may perform similar to HCCC aluminum, but has the significant drawback of increased product weight making them practically unsuitable for the needs of the users. It should be noted that increased product size also has negative environmental implications, especially with regards to material use and transportation inefficiency.

HCCC has also been successfully used as a pre-treatment step prior to a plastic overmolding process for magnesium castings, assuring that the plastic adheres to the magnesium material. Not using this pre-treatment step in the overmolding process with a bare magnesium casting causes the plastic to only partially bond to the metal.

3.0 Test History and Results

There is no collection of EMI test data available in the area of HCCC substitutes. Testing to address the specific needs of the T&M sector is very slim. A New Zealand study concluded that "there were no alternatives to Hexavalent Chromium for steel that outperform Alodine 1200 as a result of (their) trials...."

Another study concluded that after testing eight potential non-chromated pretreatment substitutes, "the only compositions that come close to matching the technical, process, cost, and flexibility of chromates are based on trivalent chromium."²

The Environmental Security Technology Certification Program (ESTCP) study on Non Chromate Aluminum Pretreaments, Phase II Interim Report concluded the following: "...any new coating application should be demonstrated and validated by field-testing for each operational environment where implementation is being considered. Only then can the complete technical performance of a coating or coating system be determined." 3

Introduction to T&M testing

¹ RoHS and WEEE Specialists (NZ) Ltd, 25 October 2005

² ESTCP Phase I report on Non-Chromate Aluminum Pretreatments, Aug 2003

³ ESTCP Phase II Interim Report on Non-Chromate Aluminum Pretreatments, Sept 2004

The T&M sector has conducted preliminary testing, evaluating the performance characteristics of HCCC substitutes. A phased preliminary test plan was developed to explore several materials, coatings, and joining methods and their contributions toward electrical resistivity and control of EMI.

Phase 1 involved DC Resistance testing. It is logical that a lower resistance coating may be more effective in creating a continuous EMI shield. The test was conducted for several substitutes, before and after simulated environmental degradation. Several well known possible substitutes were tested in multiple samples against HCCC and bare aluminium control samples. Substitutes included electrogalvanized plated steel, Alodine 4594, trivalent chromium (TCP), and iridite non-chromium process (NCP), each over aluminum. While these tests were conclusive in showing that hexavalent chromium does have a lower resistance than the substitutes, no standards or guidelines currently exist to correlate DC resistivity or radio frequency (RF) impedance to EMI shielding effectiveness.

This is further evidenced in the New Zealand study, "In the electronics industry, Chromate's low surface electrical resistance has been an advantage for conduction across mechanical joints for the purpose of shielding. However, we have not determined approximately what is an acceptable minimum level of surface electrical resistance."

Phase 2 EMI testing was designed to correlate DC resistance testing to the electromagnetic shielding effectiveness (measured in energy leakage, either conducted or radiated) of materials, coatings, and joining techniques given extreme conditions of random vibration, temperature cycling, and 10 day humidity testing. Conducted EMI test results were similar to radiated emissions results.

Additional testing

HCCC used as a pre-treatment in the plastic overmolding process of magnesium has been successfully demonstrated in production. Several sample substitute coatings were applied to the base magnesium material and then subjected to the overmolding process. These samples were compared to HCCC control samples.

Descriptions, results and conclusions of these tests follow. Phase III testing is also planned.

Phase 1, DC Resistance Testing

Fixtures were designed, built, and tested to determine the change in resistance over extreme conditions of random vibration (RV), temperature cycling (TC), and 10 day humidity (HUM) testing. These tests are commonly and effectively used to simulate the accelerate life of a product. A salt spray test was also completed for a visual demonstration of the cosmetic effects of a harsh environment.

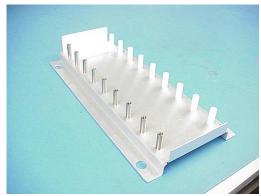


Figure 5, Empty Resistance Fixture

Figure 6, Loaded Resistance Fixture

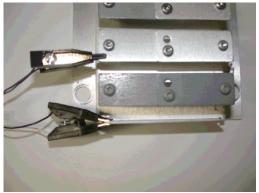


Figure 7, Resistance Measurement

Figure 8, Resistance Measurement

Material and Coatings Kev

Code	Description
Bare AL	Bare Aluminum
Cr-6	Hexavalent Chromium on Aluminum
TCP-HF	Trivalent Chromium on Aluminum
I-NCP	Iridite-NCP on Aluminum
egal	Zinc coated Steel
4594	Alodine

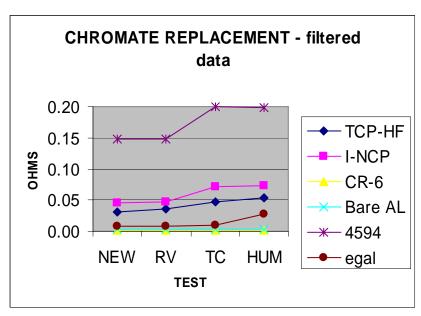


Figure 9, Resistance Testing Results

Figures 4-8 show the DC Resistance fixture and testing setup. Results in Figure 9 show elevated resistance in nearly all samples over the series of random vibration, temperature, and humidity testing. Alodine 4594 performed poorly with high DC resistance and was therefore eliminated from further testing. Cr6 and bare Aluminum showed the lowest resistance measurements for this test, both before and after environmental degradation.

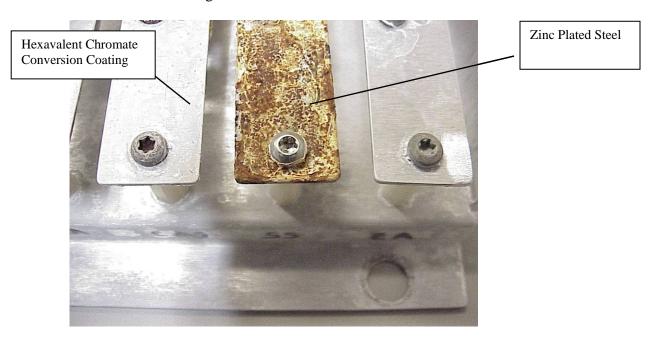


Figure 10, after Salt Spray Testing

The salt spray test (Figure 10) illustrated the obvious reaction of the test materials (Zinc Plated Steel vs HCCC) under severe conditions and a visual cosmetic concern.

Conclusions of Phase 1, DC Resistance Testing

Resistance testing showed that in the samples under test, resistance did indeed degrade under extreme conditions. This was not unexpected. However, the test results in Figure 9 are coarse and the test method does not enable the fine resolution necessary for testing a wide variety of materials. It was also unclear from this testing that resistance change is of any real material importance to controlling EMI and specific product performance.

Phase 2, EMI Testing

In Phase 2, new fixtures were designed and built to interface with an existing test bench apparatus (Figures 11-15). EMI testing (conducted and radiated) was to determine the electromagnetic shielding effectiveness (leakage) of materials, coatings, and joining techniques given extreme conditions of random vibration (RV), temperature cycling (TC), and 10 day humidity testing. Initial EMI spectrum scans were completed for each of multiple samples for "low" (25 Hz to 1 GHz) and "high" frequency (1 GHz to 5 GHz). Subsequent scans were then completed after RV and TC, and again after 5 day and 10 days humidity testing. Results were compared to the HCCC containing (control) samples. Testing above 5 GHz was not possible in the current EMI Lab configuration.



Figure 11, EMC Lab

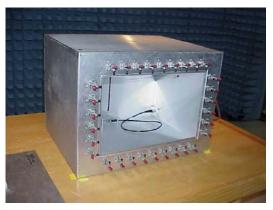




Figure 12, Bench Apparatus

Figure 13, Bench Apparatus

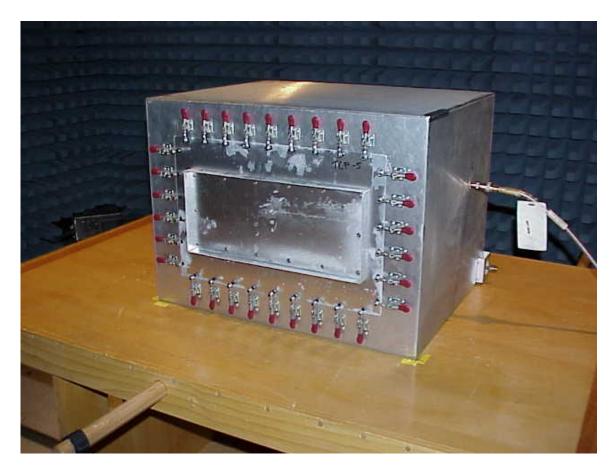
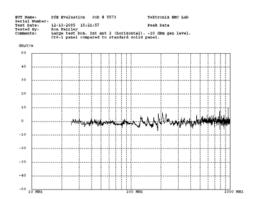


Figure 14, A Sample Fixture typically installed in the Bench Apparatus



Figure 15, EMC Lab Operational Equipment

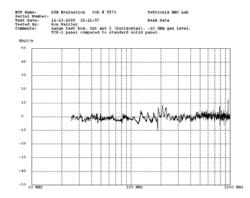


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Figure 16a, Initial Cr+6, Low Freq

Figure 16b, Initial Cr+6, Higher Freq

Figures 16a and b and 17a and b show the logarithmic electromagnetic response of several test samples over a low frequency range defined to be 25 MHz to 1 GHz and the higher frequency range of 1 GHz to 5 GHz. The materials are Cr6 and TCP, respectively. This initial test shows the response curves to be similar in both low and higher frequency leakage.



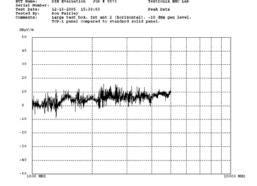


Figure 17a, Initial TCP, Low Freq

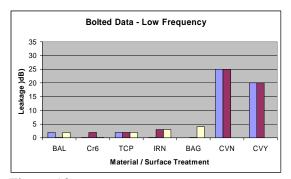
Figure 17b, Initial TCP, Higher Freq

The series of EMC response curves obtained in this testing was very difficult to analyze. An accepted method of assigning meaningful figure of merit to a response curve does not currently exist, but an attempt was nonetheless made to assign a representative "average" number to permit numerical comparison. In the following graphs, this "representative" number is shown and plotted for a number of test results.

Material and Coatings Key

Code	Description
BAL	Bare Aluminum
Cr6	Hexavalent Chromium on Aluminum
TCP	Trivalent Chromium on Aluminum
IRN	Iridite-NCP on Aluminum
GXC	Zinc coated Steel
BAG	Galvanized Steel on Aluminum
CRG	GXC bracket on Cr6 base plate
CVN	Vinyl coated Al bracket on Cr6 base plate w/nylon screws
CVY	Vinyl coated Al bracket on Cr6 base plate w/SS screws

Initial EMC Scan Results



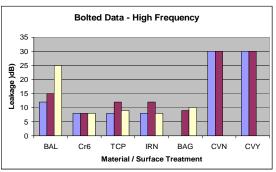
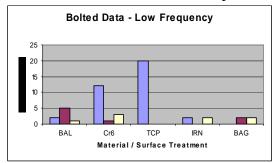


Figure 18a

Figure 18b

In Figures 18a and 18b, initial EMC scan results are nearly equivalent at low frequency for HCCC and several substitutes (TCP, IRN, and even bare aluminum). At higher frequencies, leakage is evident in all samples. RoHS compliant substitutes to Cr6 are nearly equivalent but bare aluminum has higher leakage. As expected, vinyl coated aluminum performs poorly as a shield at low and higher frequencies. Vinyl coated aluminum was therefore eliminated from further testing.

EMC Scan Results, Post RV, Temperature Cycling Tests



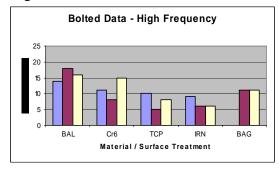


Figure 19a

Figure 19b

After random vibration and temperature cycling, the samples were tested for EMI leakage (Figures 19a and 19b above). However, screws were replaced and hand tightened in several samples. The joint was therefore presumed to be compromised. However, testing continued. Again, at higher frequencies, all samples showed more leakage.

EMC Scan Results, Post 5 day Humidity Testing





Figure 20, HCCC after 5 Day Humidity

Figure 21, TCP after 5 Day Humidity



Figure 22, Bare Al after 5 day Humidity

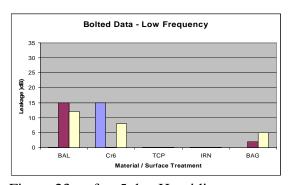


Figure 23a, after 5 day Humidity

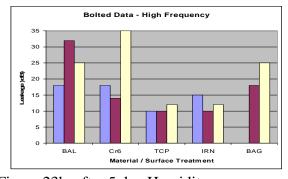


Figure 23b, after 5 day Humidity

Following 5 days of Humidity testing, the samples were removed and tested. In Figures 23a and 23b, Cr6 samples show greater leakage than TCP and IRN at both low and higher frequency.

EMC Scan Results, Post 10 day Humidity Testing





Figure 24, HCCC after 10 day Humidity

TCP-S

Figure 25, IRN after 10 day Humidity

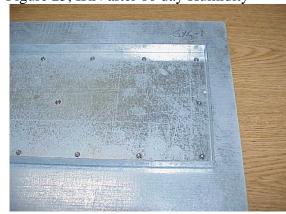


Figure 26, TCP after 10 day Humidity

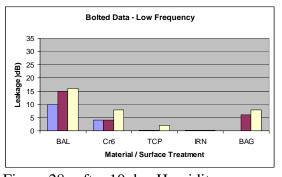


Figure 27, GXC after 10 day Humidity

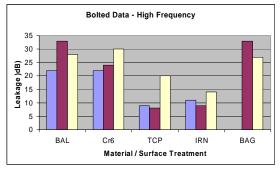


Figure 28a after 10 day Humidity

Figure 28b. after 10 day Humidity

After the full 10 day Humidity testing, Figures 28a and 28b, Cr6 again shows more leakage than TCP and IRN at both low and higher frequency.

Conclusions of Phase 2, EMC Testing

The test data from this experiment did not support the general belief that Hexavalent Chromium containing finish would be superior to the RoHS compliant finishes under

all conditions. While an important data point, the evaluation was inadequate due to the assumption of quantifying an EMC spectrum response curve (Figure 16 a and b) to a single representative number (in Figure 18 a and b) to enable comparison. There is an obvious need to address these shortcomings in the testing method in order to compare response curves. The inconsistent test results and the small sample size do not allow substantiation of a correlation between DC resistivity from Phase 1 tests and EMI emissions from Phase 2 tests.

One assumption prior to testing was that all fixtures would remain assembled and would be tested completely through the suite of tests, thus assuring that the critical contact joint would behave as in an assembled instrument. However, after RV and temperature cycling, this assumption was compromised. Several screws in certain sample sets were replaced (they loosened in the RV and temperature testing) and many screws were hand tightened, affecting the contact joint and possibly altering the test outcome. Why the screws became loose in the tests is still perplexing as all screws were originally hand tightened to a reasonably snug fit.

Nevertheless, in subsequent testing, results showed HCCC did not perform as well as several substitutes. This was surprising. Most samples performed worse at higher frequencies. All samples were cosmetically inconsistent after 5 day humidity testing. After 10 day Humidity testing, each sample exhibited worse corrosion.

Additional Testing- Magnesium Overmold Testing

Several substitute coatings (NCP, Hexavalent Chromium NH35 (control), and TCP-HF) in five samples each were tested against bare magnesium, exploring adhesion characteristics in a plastic overmolding process.



Figure 29, Mag w/HCCC overmolded (control)

The control sample of HCCC over magnesium produced sample parts similar to production in the overmolding test shot process (Figure 29).



Figure 30, NCP over Mag

Figure 31, Mag w/TCP overmolded

The supplier stopped the NCP application over magnesium in mid-process. The application was abandoned as incomplete and was not overmolded (Figure 30). The TCP surface finish over magnesium produced a completed part (Figure 31) similar to the control sample. Further environmental testing is planned to determine more long term effects.



Figure 32, bare Mag, overmolded

Figure 33, bare Mag, overmolded

Overmolding the bare magnesium illustrated why a pre-process step was initially recommended for production. Figures 32 and 33 both show an overmold that is not bonded to the magnesium part and is considered unacceptable.

Phase 3, Instrument Testing

Testing does not always proceed as planned and results obtained can be unexpected. The next testing phase includes product testing and is critical due to the inconsistency of test results from these first two testing phases. The lower DC resistance values of HCCC led us to believe that we might show this coating to be superior to substitutes over a frequency range in EMC testing. However, our testing thus far is inconclusive in showing that HCCC is more effective than other substitute coatings in controlling EMI when used on aluminum shielding. There is a clear need to reassess our experimental model to better understand test methods and control test variables.

Additional testing is planned with investigations into testing methods and functional T&M products utilizing substitute HCCC to understand effectiveness in controlling radiated and conducted EMI over a wide range of products and environments. It's very important to customers that changes in materials do not adversely affect instrument performance over the long term. Proposed material substitutes must be fully evaluated to be able to provide this level of assurance and is not taken lightly.

4.0 Summary and Future Work

Hexavalent chromate conversion coating (HCCC) on metal surfaces is a critical design element for Test & Measurement sector applications. In the area of EMI shielding our members have conducted preliminary sample testing of potential alternative material using HCCC on steel and aluminium as a baseline to establish suitability of alternatives.

Preliminary investigations and test results to date have shown:

- No independent studies have made tests on the properties of alternatives in relation to EMI shielding. DC resistivity and anti-corrosion characteristics have been the focus of such studies.
- Our own tests to 5 GHz show no direct correlation between material DC resistivity and performance at high frequencies for HCCC or alternatives.
- Our test teams have to develop a RF test methodology that is applicable to the
 test and measurement sector prior to converting our product portfolio.
 Currently, there is no recognized standard for this application of hexavalent
 chromium.
- Our tests were performed on coupon samples and have not extended to qualified product parts in their specific operating environments.
- Testing to date has not reached required upper frequencies of many Test & Measurement products that are often an order of magnitude and more above 5 GHz.
- There is no field reliability data of substitutes to HCCC validating all aspects
 of performance in respect of typical Test & Measurement product operating
 environments.

Coalition members will continue investigations to define suitability of HCCC alternatives. We estimate at minimum another four years is needed for thorough technical evaluation of potential substitutes. In addition we have to quantify needs, if any, for manual handling of substitutes when they have been proven to meet technical requirements. For example, will absence of surface self-healing associated with HCCC raise additional issues to resolve in practical use of a substitute? Only then will we be in a position to confirm if suitable substitutes can be realized successfully across our varied product families.

As part of the verification of any substitute materials, the following testing will be planned and executed:

- development of a validation methodology for the testing;
- alternate test methods allowing investigation of other possible coating substitutes;
- other material parameters, electrical and magnetic shielding response at higher frequencies;
- experiments, particularly involving actual products rather than material samples, to resolve critical questions;
- extensive accelerated life experiments which, due to the longevity of T&M equipment, require several years to perform. ¹

5.0 Exemption Request

The Test & Measurement sector of the monitoring and control industry requests an exemption for the use of hexavalent chromate conversion coating in Test & Measurement products for four years after T&M products are brought into the scope of the RoHS Directive (estimated to be four years after the initial RoHS Directive requirements come into force), since there is no technically viable proven alternative which is known to meet the performance requirements in our sectors' products. This exemption will be subject to the RoHS-mandated periodic review of evidence to determine whether substitute materials are commercially available.

Phase-in Period

A transition phase-in period of four years from inclusion of T&M products in the scope of RoHS to substitute any other use of hexavalent chrome in our products. This period is considered realistic to conduct supplier surveys and replace any instances found in other applications of hexavalent chrome.